

REPORT DOCUMENTATION

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AD-A238 470

Public reporting burden for this collection of information is estimated to average 1 hour gathering and maintaining the data needed, and completing and reviewing the collection of information, including suggestions for reducing this burden, to Washington Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management

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20503

1. AGENCY USE ONLY (Leave blank)

2. REPORT DATE

2/27/91

4. TITLE AND SUBTITLE

The Microstructure and Corrosion Resistance of Ion Implanted Aluminum-Molybdenum Alloys Containing the Phase Al₁₂Mo

5. FUNDING NUMBERS

DAAG29-78-G-0022

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DAAG29-79-D-1003

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

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8. PERFORMING ORGANIZATION
REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

U. S. Army Research Office
P. O. Box 12211
Research Triangle Park, NC 27709-2211

10. SPONSORING/MONITORING
AGENCY REPORT NUMBER

ARO 15489.3-M5

ARO 18694.1-M5

11. SUPPLEMENTARY NOTES

The view, opinions and/or findings contained in this report are those of the author(s) and should not be construed as an official Department of the Army position, policy, or decision, unless so designated by other documentation.

12a. DISTRIBUTION / AVAILABILITY STATEMENT

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12b. DISTRIBUTION CODE

13. ABSTRACT (Maximum 200 words)

For solid solution iron base alloys with chromium formed by either ion implantation or bulk melting, the corrosion behavior in aqueous chloride solution can be represented by potentiodynamic polarization measurements. This was established by comparing the potential-pH diagrams obtained in this study to published Pourbaix diagrams of potential versus pH for the appropriate systems.

Analytical electron microscopy revealed that continuous films of the phase Al₁₂Mo with either a mottled, metastable microstructure or a granular, stable microstructure formed in aluminum annealed at selected temperatures after implantation with a maximum molybdenum concentration in the range of 8 to 10 atomic percent. The corrosion behavior of specimens containing either of these two types of continuous films or an as-implanted microstructure was characterized in aqueous solutions with or without 0.1 molar chloride ion at pH6 or pH6.3 using potentiodynamic polarization techniques. The corrosion behavior in the selected environments depended on the microstructure and distribution of both molybdenum and oxygen in the near surface region. A model for the corrosion behavior was developed that was consistent with the experimental results.

14. SUBJECT TERMS

Iron-chromium alloys,
Corrosion Behavior

15. NUMBER OF PAGES

24

16. PRICE CODE

17. SECURITY CLASSIFICATION
OF REPORT

UNCLASSIFIED

18. SECURITY CLASSIFICATION
OF THIS PAGE

UNCLASSIFIED

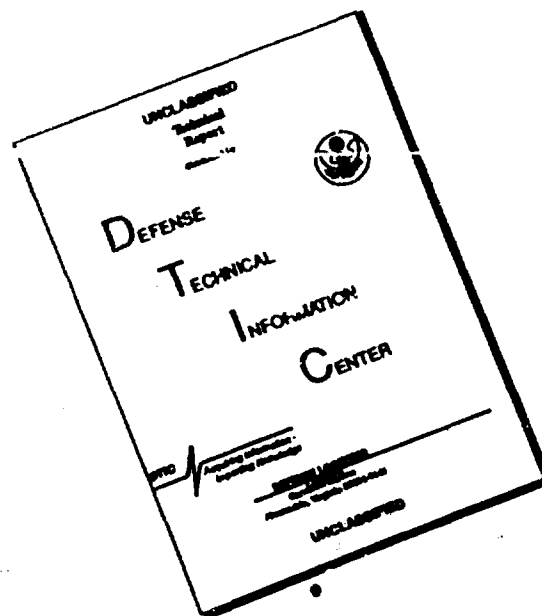
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20. LIMITATION OF ABSTRACT

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**The Microstructure and Corrosion Resistance of Ion
Implanted Aluminum-Molybdenum Alloys
Containing the Phase Al₁₂Mo**

Final Report

Ray B. Benson, Jr.

U.S. Army Research Office

Contract Number DAAG 29-79-D-1003

DAAG 29-78-G-0022

North Carolina State University

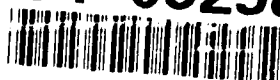
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Statement of the Problem

The work summarized in this report falls in several areas. The first part deals with determining experimental aqueous chloride potential-pH diagrams using potentiodynamic polarization techniques for pure iron, iron five weight percent chromium bulk alloys, and iron implanted with an average chromium concentration of eight percent in the surface modified region. These diagrams were compared with each other and to available equilibrium Pourbaix diagrams for the appropriate systems in order to help evaluate the use of ion implanted alloys to obtain corrosion data for bulk alloys with the same chemical composition.

The microstructure in the surface modified region of aluminum annealed after implantation with a range of maximum molybdenum concentrations were examined in both the as-implanted and as-implanted-annealed states using transmission electron microscopy techniques. After annealing for a certain range of maximum molybdenum concentration, a continuous film of the phase $Al_{12}Mo$ formed in the surface modified region with both a metastable and stable microstructure.

In the third part of this report, the corrosion properties, as determined by potentiodynamic polarization techniques, of alloys containing these two different types of continuous $Al_{12}Mo$ films in the surface modified region are summarized.

Summary of the Results

1.0 Corrosion Behavior of Iron-Chromium Alloys

The corrosion behavior of Aramco iron, a bulk Aramco iron-5w/o chromium alloy, and an Aramco iron base alloy with an average as-implanted chromium concentration of approximately 8 w/o were compared in an aqueous solution with and without 0.1 molar chloride ion in the pH range of 2 to 12 by examining the experimental potential -pH diagrams constructed from potentiodynamic polarization measurements [1,2]. One of the

goals of this study was to help evaluate the usefulness of representing alloy corrosion behavior by experimental potential-pH diagrams determined in this way. Another goal was to evaluate the corrosion behavior of bulk alloys using ion implanted alloys with the same approximate surface solute concentration as the bulk alloy.

The experimental potential-pH diagram for pure iron (Fig. 1) is in good agreement with the equilibrium Pourbaix diagram for the iron-water system [3] which is a good indication this is a useful method for representing the corrosion behavior of metals. The effect of chloride ion on the potential -pH diagram for pure iron in aqueous solution is to reduce the passive region for pitting as is seen by comparing Fig. 1 and Fig. 2. The effect of chromium implantation on the experimental potential -pH diagram for an aqueous solution without chloride ions, as illustrated in Fig. 1 and Fig. 3, is to shift the corrosion potential to a more noble potential than that of the unimplanted iron and to extend the passive region from pH7 to pH4.

For the 0.1 molar chloride solution, as can be observed by comparing Fig. 1 and Fig. 4, the major difference between the Aramco iron and the iron implanted with chromium is that the range of passive film stability is limited to pH6 at the lower pH limit for the chromium implanted alloy. The implanted alloy is immune to corrosion at potentials more noble than the unimplanted alloy, but the improvement is limited to pH less than 8. The pitting potential is essentially the same for both the implanted and unimplanted alloy in the presence of chloride ion.

A comparison of the overall corrosion behavior in 0.1 molar chloride solution of the chromium implanted alloy (Fig. 4) with the bulk alloy containing a similar chromium composition (Fig. 5) reveals the corrosion behavior of both alloys is very similar, except that the passivation range of the implanted alloy is limited to pH 6-14, whereas, the passive film on the conventional iron-chromium alloy is stable in the range extending to pH 3.5. It appears that the major enhancement of corrosion behavior by chromium implantation is due to the implanted surface chemistry and not an effect due to ion damage.

The results of this investigation indicate (1) experimental potential -pH diagrams determined from potentiodynamic polarization measurements are useful for representing general and pitting corrosion behavior for alloys in aqueous solution, and (2) the corrosion behavior of bulk alloys in aqueous chloride solutions can be represented by ion implanted alloys with the same average solute composition in the surface modified region as bulk alloys.

2.0 Microstructure of Aluminum Annealed After Implantation with Molybdenum Ions

The microstructure formed in aluminum annealed after implantation with a range of molybdenum concentrations was examined using selected analytical electron microscopy techniques and Rutherford backscattering spectroscopy [4-11]. Dual 50-100 KeV implant energies were employed in order to produce a broader surface modified region for formation of these microstructures. For each alloy in the as-implanted state, a crystalline material containing ion damage was observed using TEM techniques. When aluminum was annealed at 550°C for two minutes after implantation with maximum molybdenum concentrations in the range of 2.5 to 3.5 atomic percent (a/o), a lamellar $Al_{12}Mo$ precipitate structure (Fig. 6a) was observed throughout the surface modified region [8,11]. An interlocking $Al_{12}Mo$ precipitate structure (Fig. 6b) formed throughout the near surface region when specimens with the same molybdenum concentration range were annealed at 550°C for one and a half hours [8,11].

For aluminum annealed after implantation with molybdenum in the approximate concentration range of 8 to 11 a/o, a continuous film of $Al_{12}Mo$ formed in the surface modified region. The microstructure of the continuous $Al_{12}Mo$ film formed depended on the annealing temperature. A metastable $Al_{12}Mo$ film with a mottled microstructure (Fig. 7a) formed on annealing at 400°C for forty eight hours [8]. A stable film of $Al_{12}Mo$ grains (Fig. 7b) formed during annealing at 550°C for one and a half hours [8]. Selected area diffraction and convergent beam electron diffraction analysis along with molybdenum

concentration profiles determined by Rutherford backscattering spectrometry all indicated the Al_{12}Mo films were continuous. The molybdenum concentration of all the films determined by x ray microanalysis was also consistent with the films being Al_{12}Mo [9]. Selected area diffraction and convergent beam electron diffraction analysis indicated the crystal structure of the continuous precipitate films were the same as reported for bcc Al_{12}Mo [9]. Sequential annealing of specimens at 550°C for one and a half hours followed by an anneal at 400°C for forty eight hours produced the metastable Al_{12}Mo continuous film followed by the stable Al_{12}Mo continuous film [10].

3.0 The Corrosion Behavior of Specimens Containing Continuous Al_{12}Mo Films

The corrosion behavior of specimens containing the continuous Al_{12}Mo film with either the mottled, metastable or granular, stable microstructure in the surface modified region was characterized in an aqueous solution with and without 0.1 molar chloride ion at pH 6 and 3 using potentiodynamic polarization measurements [13]. Dual energy molybdenum implants were carried out at either 25 KeV followed by 95 KeV or at 95 KeV followed by 25 KeV. The lower energy implant was done at 25 KeV in order to have a high molybdenum concentration as close as possible to the specimen surface. For the potentiodynamic-polarization measurements, the variation in specimen current at the anodic specimen surface as the potential on the specimen is varied indicates what type of process is occurring at the anodic specimen surface. Correlations between the metallurgical microstructure, composition profiles, and corrosion behavior were explored. A model was developed to explain the corrosion behavior of aluminum implanted with molybdenum in 0.1 molar aqueous chloride solution that was consistent with the experimental evidence [13].

Pure aluminum owes its general corrosion resistance to the formation of an aluminum oxide film at pH6 (Fig. 8) but suffers pitting attack in 0.1 molar chloride solution at the same pH (Fig. 9). Aluminum as-implanted with molybdenum exhibits a

more noble corrosion potential than pure aluminum and exhibits a protective molybdenum oxide film without chloride ion present at both pH6 (Fig. 8) and pH3. This film is hard to break down in chloride solution at pH6 (Fig. 9 or Fig. 10) but is unstable in a solution containing chloride ion at pH3 (Fig. 11). Auger spectroscopy indicates there is a high molybdenum concentration adjacent to the ion implanted surface in the as-implanted specimens and that the molybdenum concentration in the near surface region increases during corrosion in 0.1 molar chloride solution at pH6.

Annealed specimens containing a continuous Al_{12}Mo film in the surface modified region with both types of film microstructures have good corrosion resistance in solutions without chloride ions at both pH6 (Fig. 8) and pH3 due to the formation of a molybdenum oxide passive film. The Auger spectroscopy results of Fig. 12 where the (Mo/Al) and (O/Mo) concentration profiles are plotted indicate that a molybdenum oxide film exists between the surface and the continuous Al_{12}Mo film after the specimens are annealed at both annealing temperatures.

Annealed specimens exhibit different types of corrosion behavior in chloride solution which depends on the dual energy molybdenum implant sequence, the type of microstructure in the continuous film which is determined by the annealing conditions, and the pH of the solution. Specimens annealed at 400°C for forty eight hours after implantation with a sequence of 25 → 95 KeV molybdenum ions (Fig. 9) or 95 → 25 KeV molybdenum ions (Fig. 10) to produce a metastable, mottled continuous film microstructure exhibited a considerable degree of passivation with respect to pitting corrosion in a 0.1 M chloride solution at pH6. A specimen annealed at 550°C for one and a half hours after implantation with molybdenum by a 25 → 95 KeV energy sequence exhibited some passivation in a 0.1 M chloride solution at pH6 [Fig. 9], whereas a specimen annealed at the same conditions after a 95 → 25 KeV energy implant sequence did not exhibit any passivation in the same solution (Fig. 10).

An atomic number analytical electron microscopy (AEM) technique where the Rutherford backscattering image from molybdenum atoms is examined in the transmission mode indicates a nonuniform Al_{12}Mo film thickness in specimens annealed at 400°C for forty eight hours for both implant energy sequences. However, for the specimen annealed at 550°C for one and a half hours after an implant sequence of $95 \rightarrow 25$ KeV (i.e. granular film structure), the continuous Al_{12}Mo film appears to be of uniform thickness. Both the as-implanted and as-implanted-annealed molybdenum concentration profiles from Rutherford backscattering measurements are consistent with these interpretations of the continuous film thickness [13].

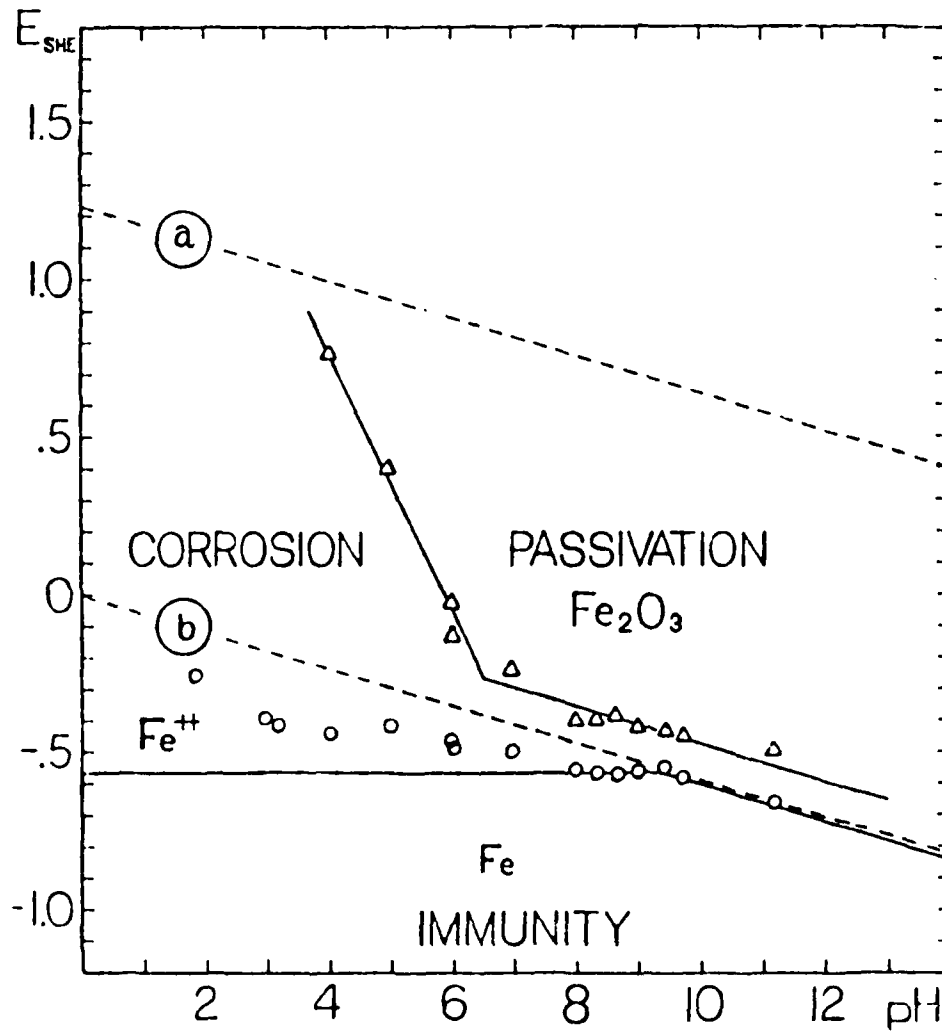


Figure 1. Experimental potential - pH diagram for unimplanted iron in aqueous solution without chloride.

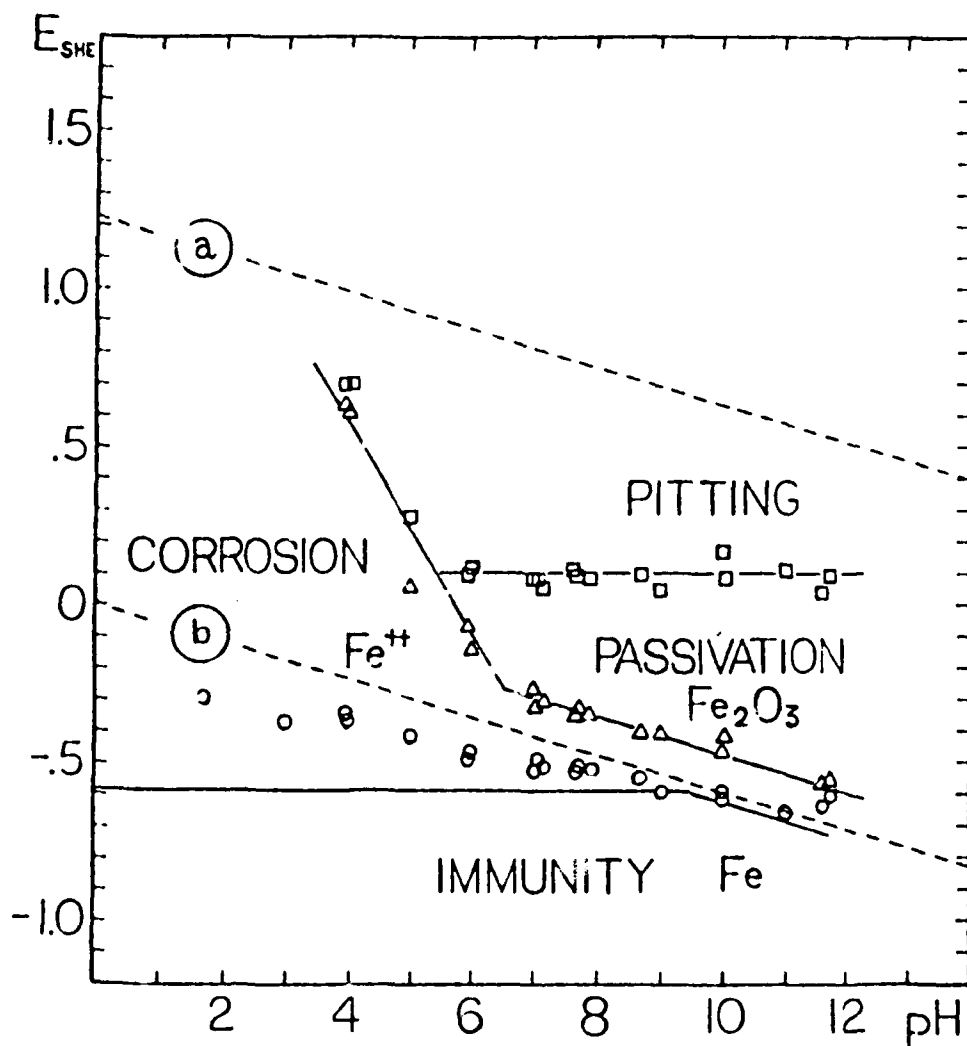


Figure 2. Experimental potential - pH diagram for unimplanted iron in a 0.1 molar chloride solution.

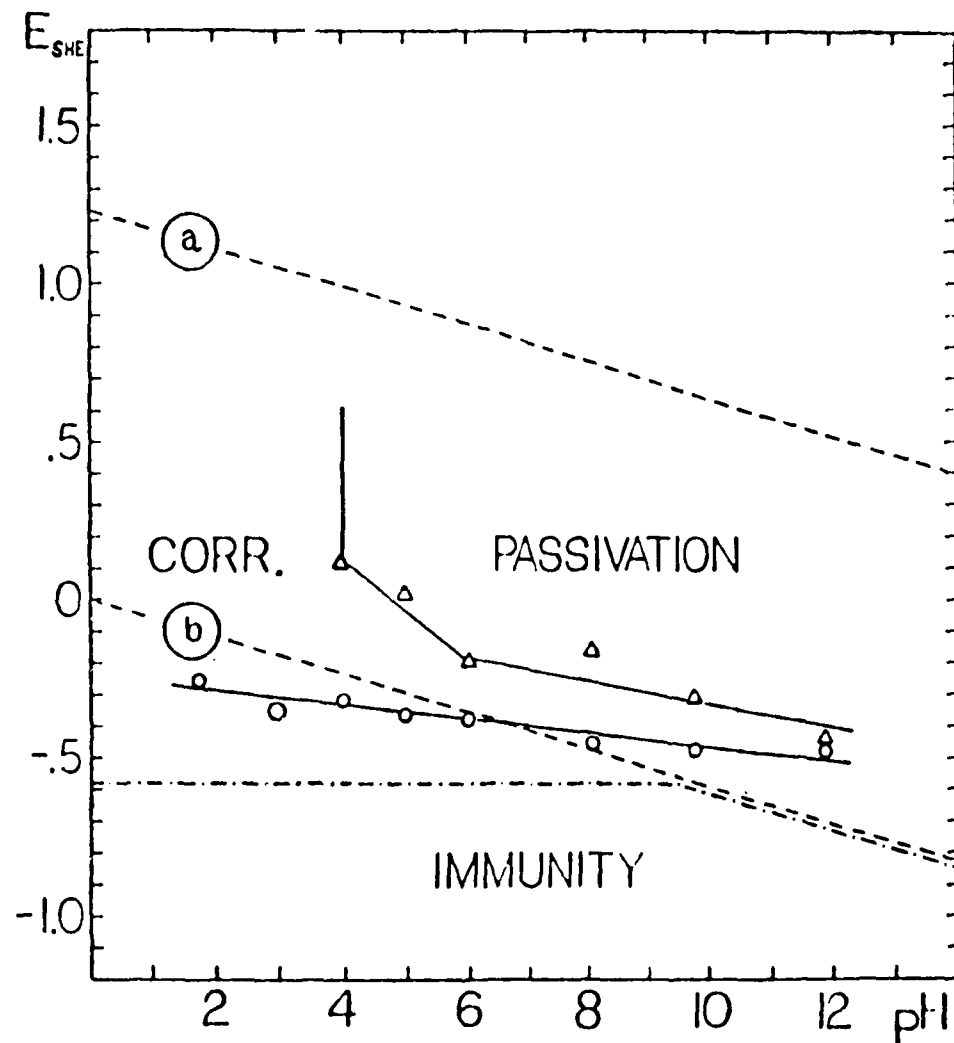


Figure 3. Experimental potential - pH diagram for iron implanted with chromium in aqueous solution without chloride.

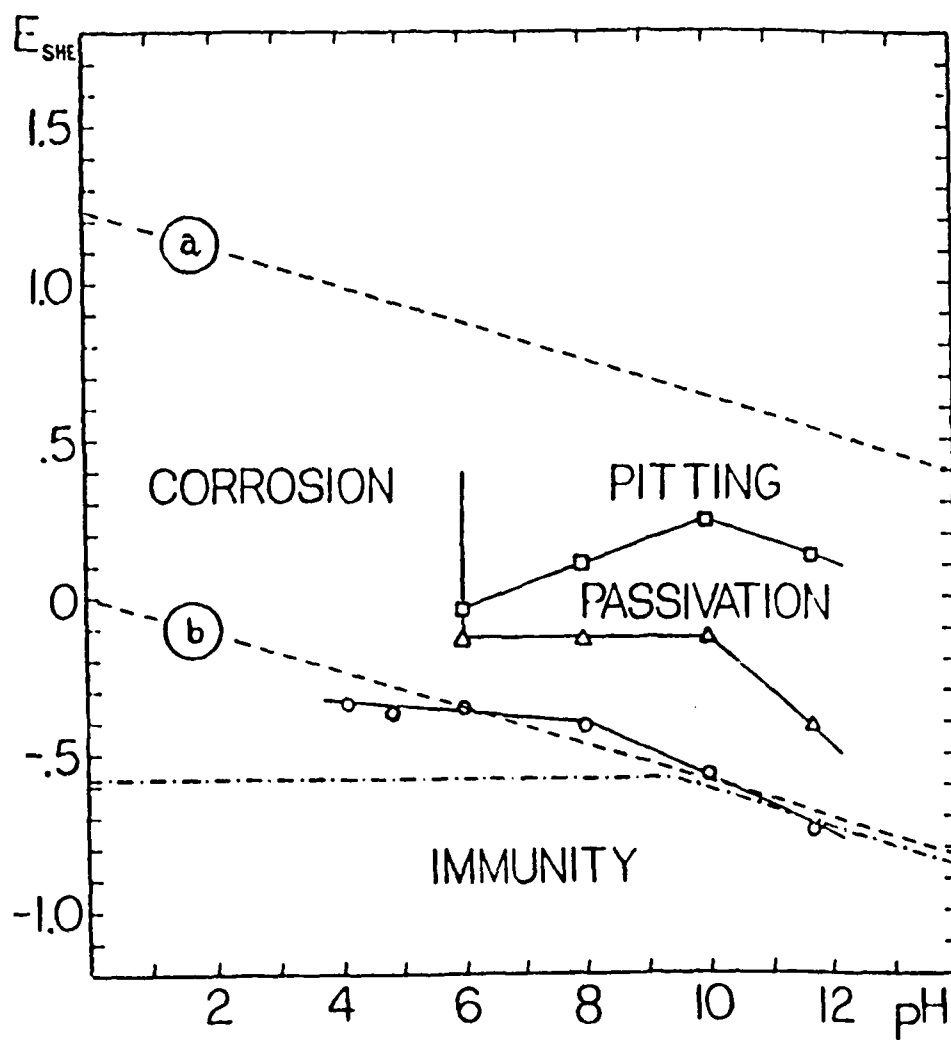


Figure 4. Experimental potential - pH diagram for iron implanted with chromium in 0.1 molar chloride solution.

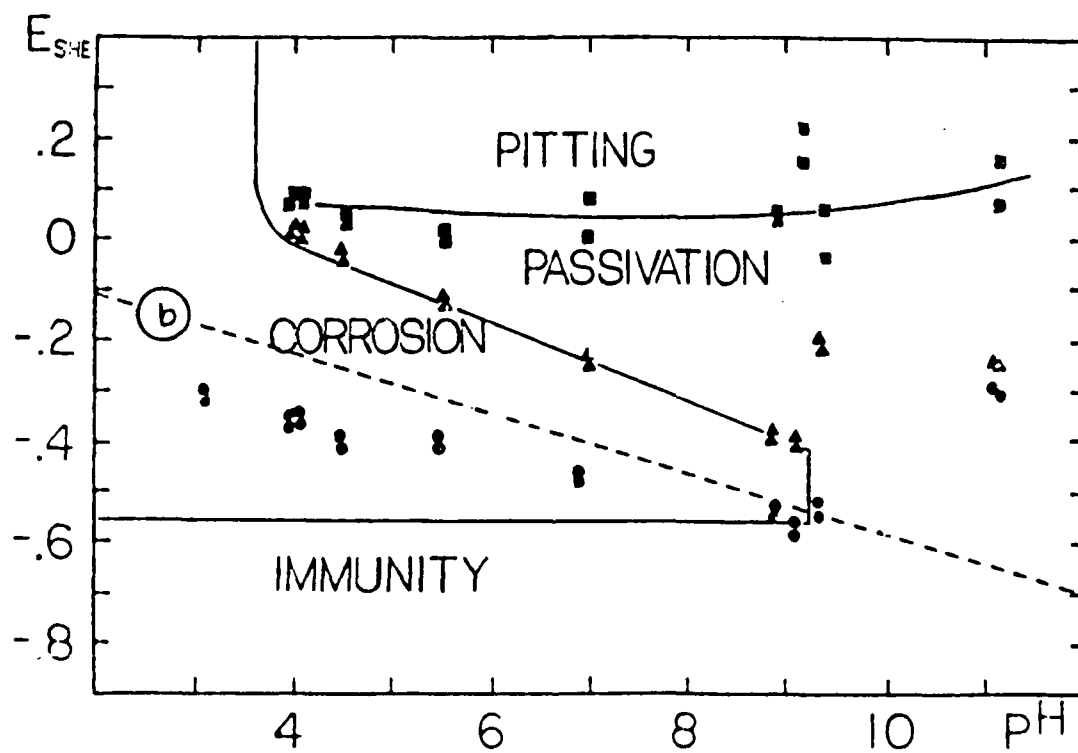


Figure 5. Experimental potential - pH diagram for bulk iron-five weight percent chromium alloy in 0.1 molar chloride solution.

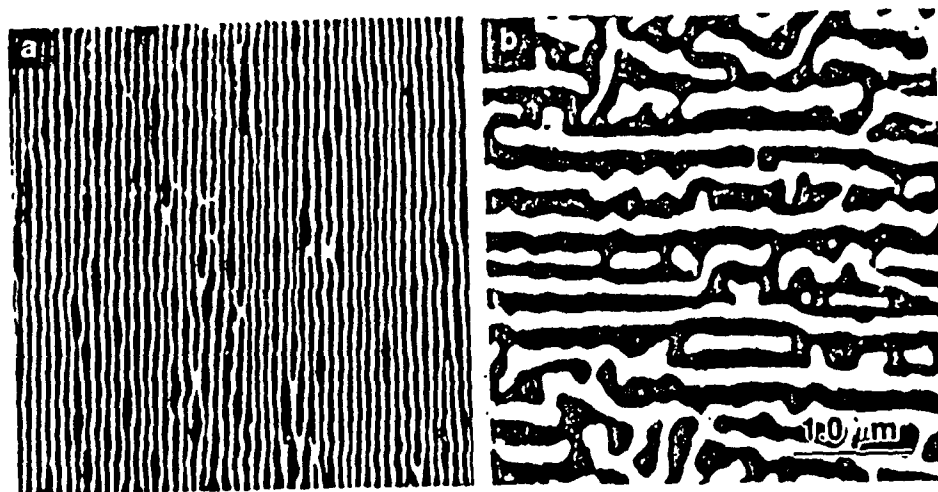


Figure 6. Precipitate morphologies in aluminum implanted with molybdenum to maximum concentration in the range of 3.3 - 4.4 atomic percent: (a) lamellar morphology in specimen annealed at 550°C for two minutes, (b) interlocking lamellar morphology in specimen annealed at 550°C for ninety minutes.

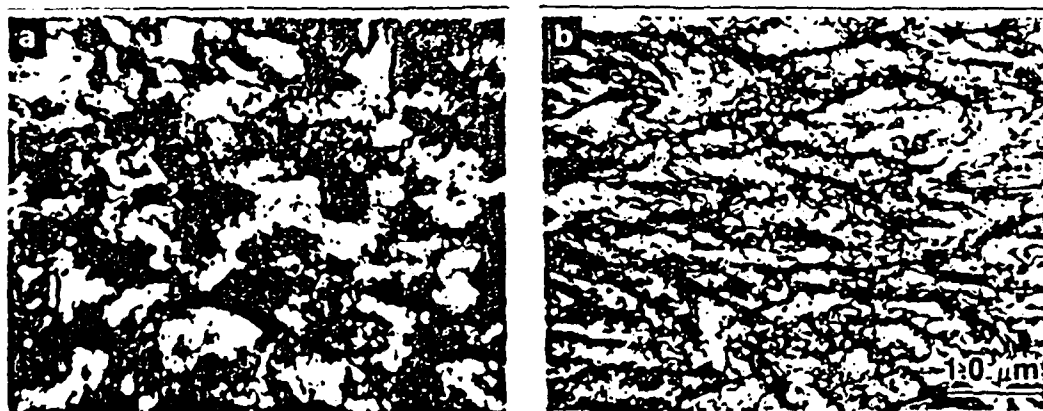


Figure 7. Microstructure of continuous Al_2Mo films in aluminum annealed after implantation with molybdenum to maximum concentrations in the range of 8 to 11 atomic percent: (a) granular stable structure formed by annealing at 550°C for one and a half hours, (b) mottled metastable structure formed by annealing at 400°C for forty-eight hours.

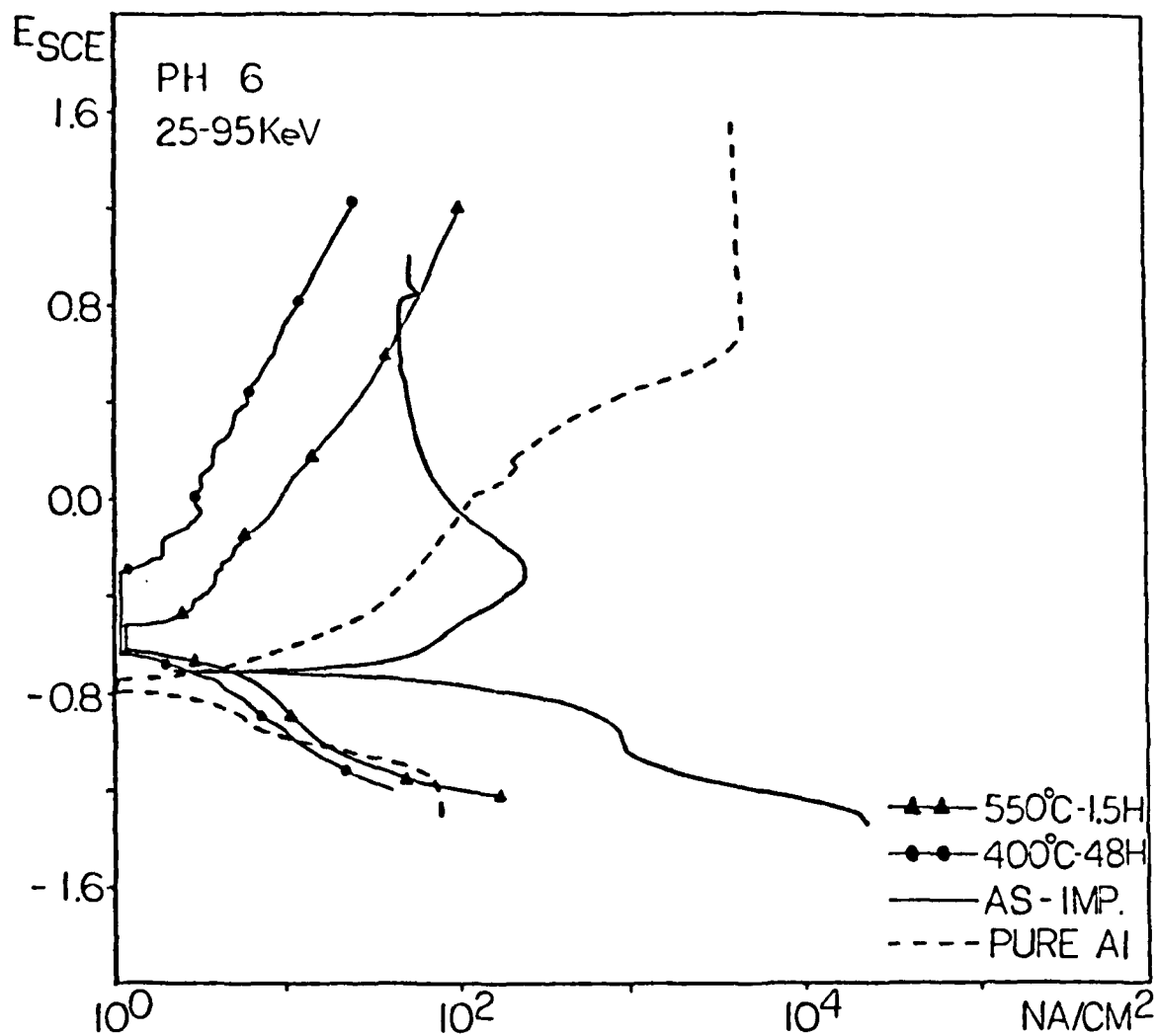


Figure 8. Potentiodynamic corrosion behavior of aluminum annealed after implantation with molybdenum (25-95 KeV) in the range of 8 to 11 atomic percent in a pH6 aqueous solution without chloride.

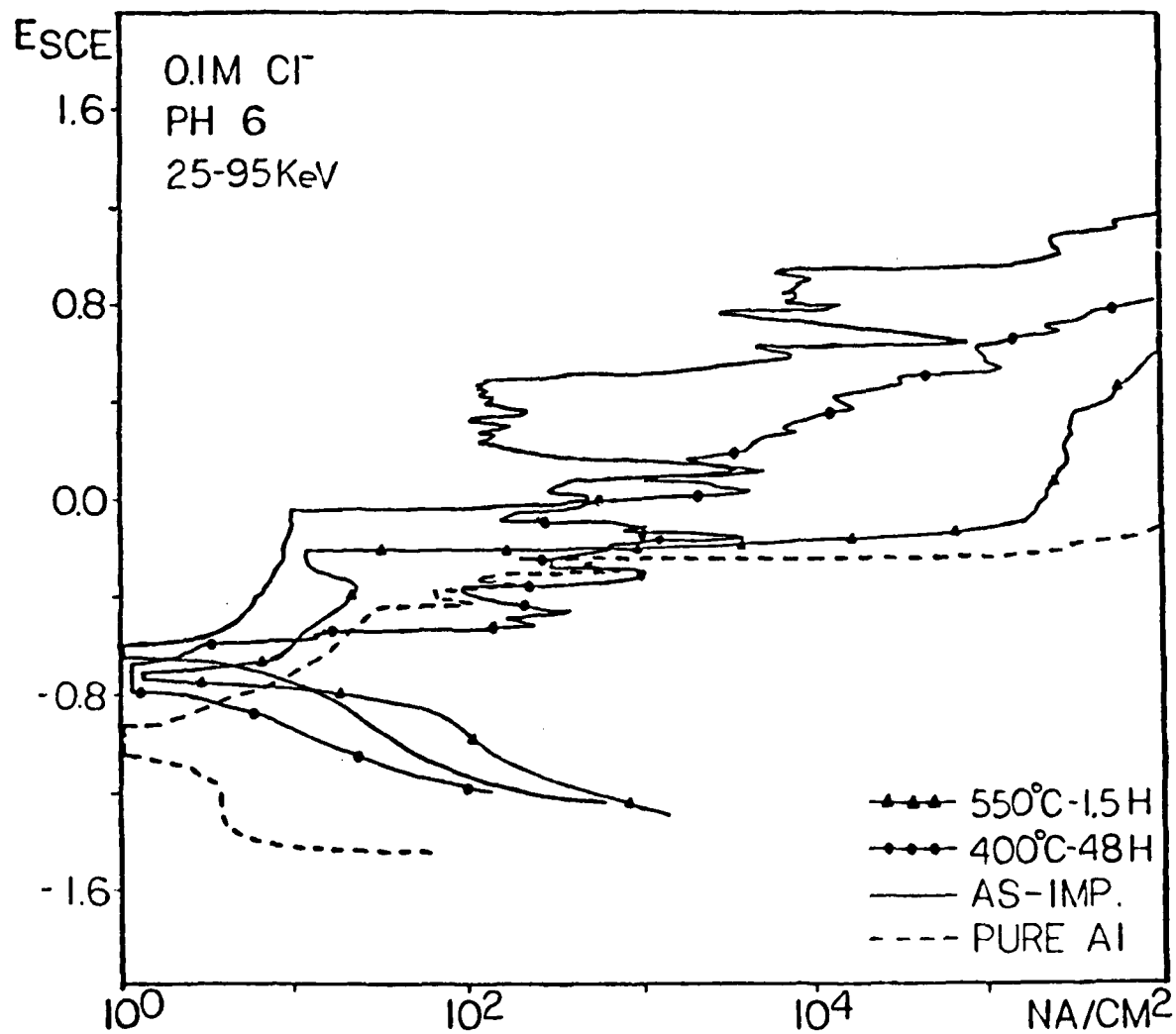


Figure 9. Potentiodynamic corrosion behavior of aluminum annealed after implantation with molybdenum (25-95 KeV) in the range of 8 to 11 atomic percent in a pH6 aqueous solution with 0.1M chloride.

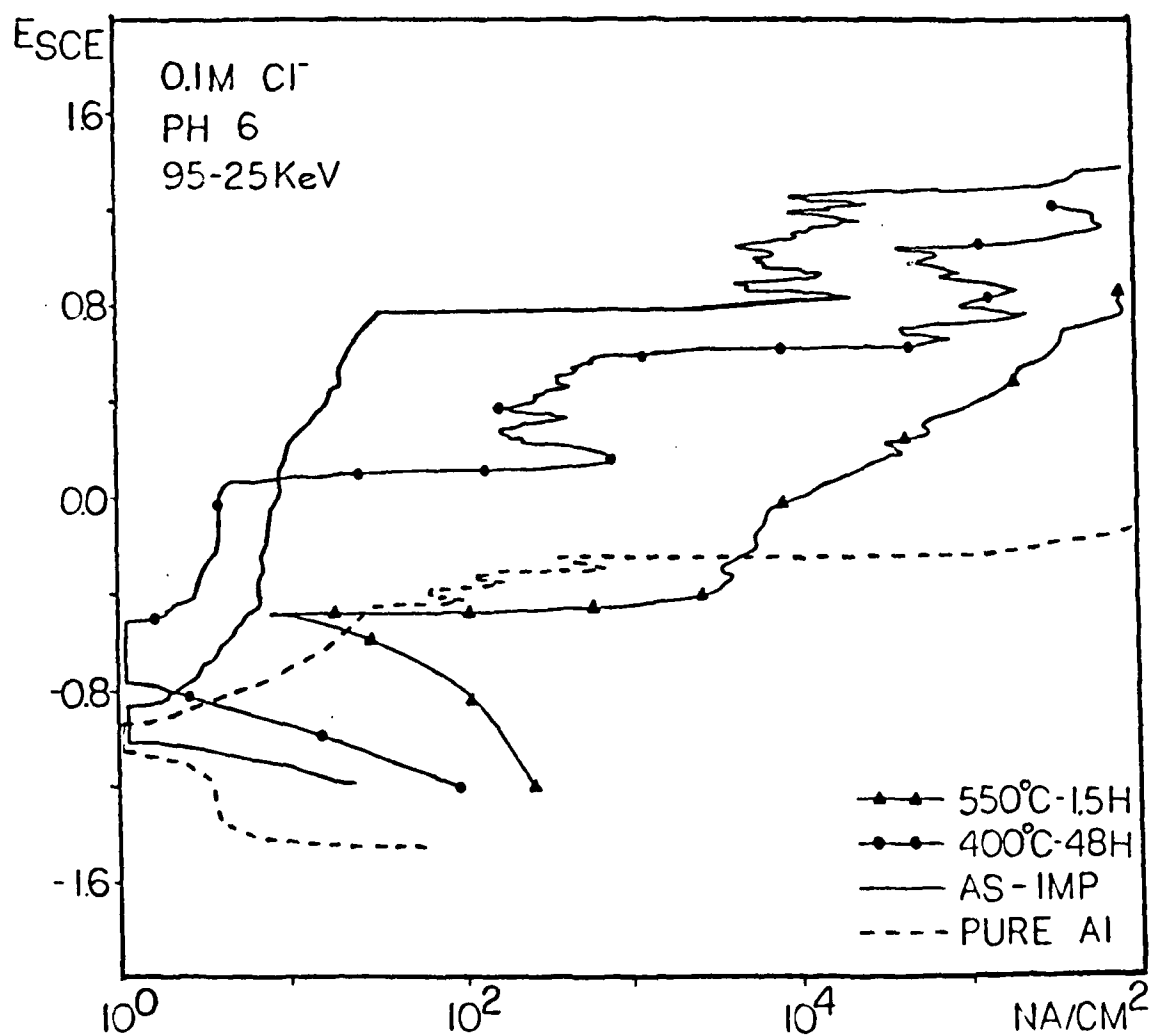


Figure 10. Potentiodynamic corrosion behavior of aluminum annealed after implantation with molybdenum (95-25 KeV) in the range of 8 to 11 atomic percent in a pH6 aqueous solution with 0.1M chloride.

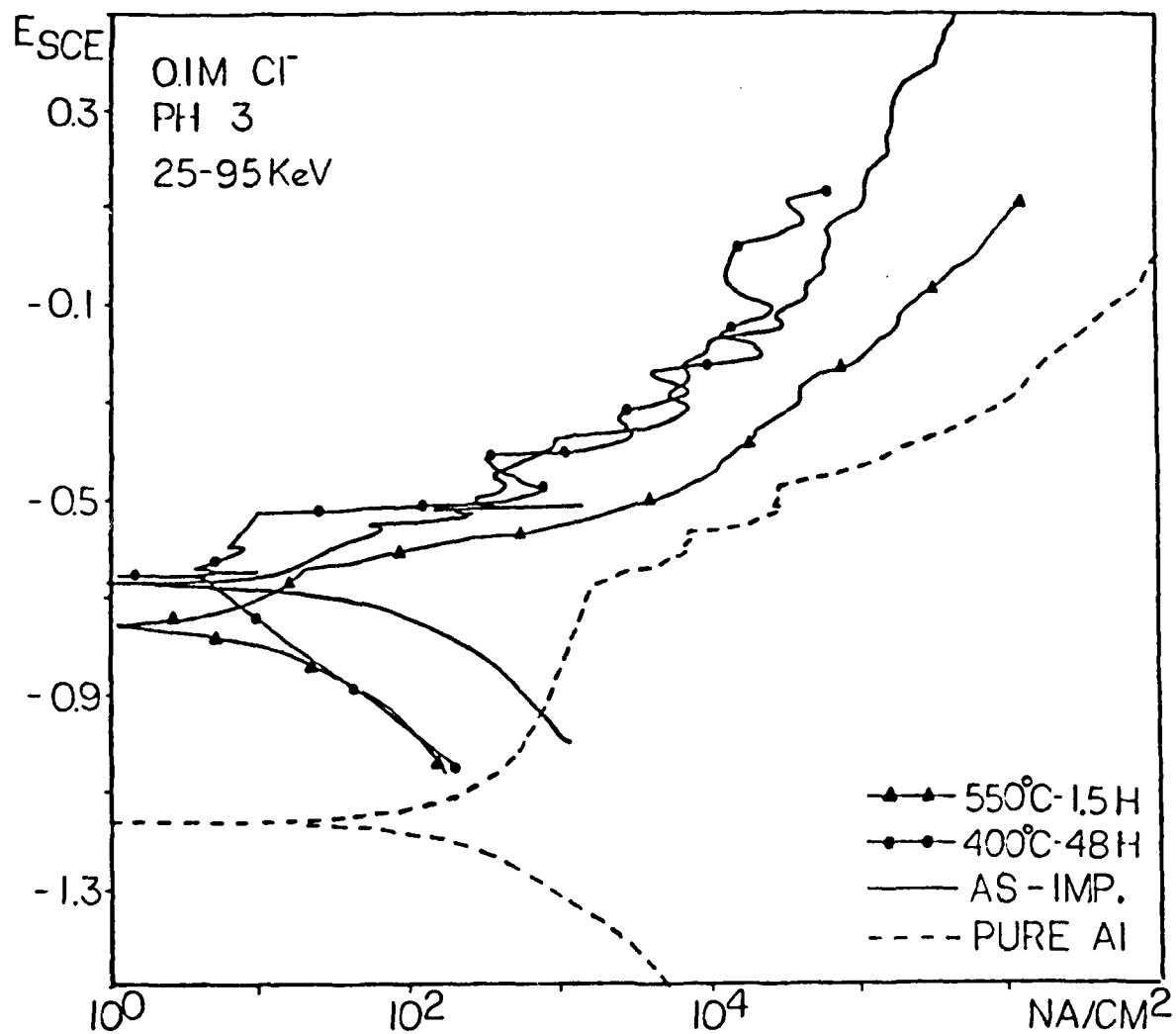


Figure 11. Potentiodynamic corrosion behavior of aluminum annealed after implantation with molybdenum (25-95 KeV) in the range of 8 to 11 atomic percent in a pH3 aqueous solution with 0.1M chloride.

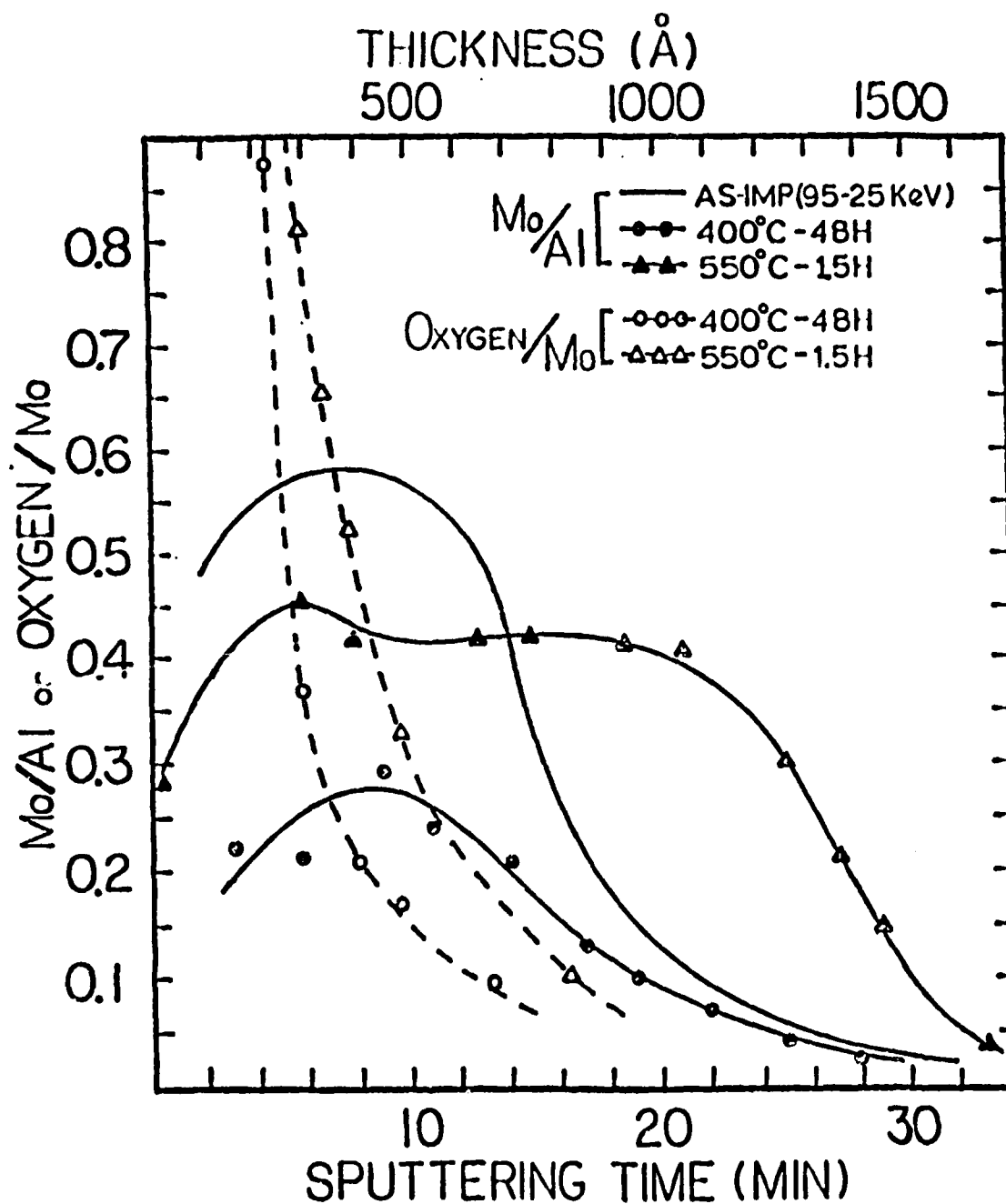


Figure 12. Auger electron spectroscopy profiles for aluminum annealed after implantation with molybdenum in the range of 8 to 11 atomic percent.

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7. J. Bentley, L. D. Stephenson, R. B. Benson, Jr., P. A. Parrish, and J. K. Hirvonen, "Second Phase Formation in Aluminum Annealed After Ion-Implantation with Molybdenum", in Ion-Implantation and Ion Beam Processing of Materials, eds., G. K. Hubler, O. W. Holland, C. R. Clayton, and C. W. White (North Holland, Amsterdam, Netherlands, 1984) p.151.
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10. L. D. Stephenson, J. Bentley, R. B. Benson, Jr., G. K. Hubler, and P. A. Parrish, "Some Characteristics of $Al_{12}Mo$ in Aluminum Annealed after Implantation with Molybdenum", in Beam Solid Interactions and Phase Transformation, eds. H. Kuary, G. L. Olsen and J. M. Poate. (Matls. Res. Soc., Pittsburgh, 1986) p. 461.

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List of Publications

1. P. A. Parrish, S. Kim, and R.B. Benson, Jr., "Effect of Chromium Implantation on the Electrochemical Behavior of Iron," in Ion Implantation into Metals, eds., V. Ashworth, W.A. Grant, and R.P. M. Proctor (Pergamon Press, New York, 1982) p. 12.
2. L. D. Stephenson, J. Bentley, R. B. Benson, Jr., and P. A. Parrish, "Analytical Electron Microscopy of Aluminum Ion-Implanted with Molybdenum, in Proceedings of the Forty-first Annual Meeting of the Electron Microscopy Society of America, ed., G. W. Bailey (Claitors, Baton Rouge, LA 1983) p.262
3. J. Bentley, L. D. Stephenson, R. B. Benson, Jr., and P. A. Parrish, "In Situ Annealing of Aluminum Ion-Implanted with Molybdenum", Proceedings of the Forty-first Annual Meeting of the Electron Microscopy Society of America, ed., G. W. Bailey (Claitors, Baton Rouge, LA, 1983) p.260.
4. J. Bentley, L. D. Stephenson, R. B. Benson, Jr., P. A. Parrish, and J. K. Hirvonen, "Second Phase Formation in Aluminum Annealed After Ion-Implantation with Molybdenum", in Ion-Implantation and Ion Beam Processing of Materials, eds., G. K. Hubler, O. W. Holland, C. R. Clayton, and C. W. White (North Holland, Amsterdam, Netherlands, 1984) p.151.
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7. L. D. Stephenson, J. Bentley, R. B. Benson, Jr., G. K. Hubler, and P. A. Parrish "Some Characteristics of $Al_{12}Mo$ in Aluminum Annealed after Implantation with Molybdenum", in Beam Solid Interactions and Phase Transformation, eds. H. Kuari, G. L. Olsen and J. M. Poate. (Matls. Res. Soc., Pittsburgh, 1986) p. 461.
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9. R.B. Benson, Jr., Invited paper, "Microstructure Control in Surface Modified Materials Using Prototype Systems", in Ion Implantation 1988, ed. F.H. Wohlbier (Trans. Tech. Publications Ltd., Aedermannsdorf, Switzerland, 1988) p. 127.

List of Participating Personnel with Advanced Degrees Obtained:

1. S. Kim
 - a) Enhanced Corrosion Resistance of Metal Surfaces by Ion Implantation (M.S. Thesis, North Carolina State University, 1980).
 - b) The Relationship Between Chemical Composition, Microstructure and Corrosion Properties in Molybdenum Ion Implanted Aluminum (Ph.D. Thesis, North Carolina State University, 1986).
2. L.D. Stephenson

An Investigation of Microstructures in Aluminum Implanted with Molybdenum Ions (Ph.D. Thesis North Carolina State University, 1984).